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Activated Sludge Treatment of Selected Aqueous Organic Hazardous Waste Compounds

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ACTIVATED SLUDGE TREATMENT OF SELECTED AQUEOUS ORGANIC HAZARDOUS WASTE COMPOUNDS

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INTRODUCTION

As a result of the Hazardous and Solid Waste Amendments of 1984 and the concurrent land disposal restrictions rule, EPA is in the process of demonstrating achievable treatment techniques to be used as alternatives to the land disposal of hazardous wastes. Data are being collected for this purpose from three types of sources, in the following order of priority: (1) waste generators that also treat the waste; (2) commercial facilities (other than generators) and (3) EPA in-house research treatment facilities. The work being done at the USEPA Test & Evaluation (T&E) Facility falls in the third category.

The USEPA, in conjunction with the University of Cincinnati College of Engineering, conducts a variety of pilot and bench scale waste treatment research projects at the T&E Facility. The purpose of several of the current projects is to develop the capability of evaluating the applicability of proposed treatment methods to both inorganic and organic hazardous wastes. The inorganic hazardous wastes to be tested are primarily metals and cyanide-bearing wastes while the organic wastes include spent halogenated and non-halogenated solvents as well as wastes from still bottoms or process wastewaters.

For aqueous spent solvent wastes containing contaminants in concentrations up to 10,000 ppm, the activated sludge process has been proposed as a potential applicable treatment technology. However, these solvent contaminants may be present in the wastes at concentrations toxic to the activated sludge biomass or they may be easily stripped into the atmosphere, creating an environmental hazard. Likewise, many of the contaminants may readily be associated with the resultant waste sludges due to sorption, precluding land disposal of the sludge. It is also possible that they may be recalcitrant and require a different treatment approach. For these reasons, the removal mechanisms for the contaminants must be assessed. Evaluation of the fate of the contaminants at several concentrations spanning the range of solubility of the compounds is also desirable.

Prior to testing industrially generated hazardous wastes, two prototype bench-scale activated sludge systems have been set up and are being used to evaluate the fates of two selected aqueous organic hazardous waste constituents, methyl ethyl ketone (MEK) and 1,1,1-trichloroethane (TCA). Both compounds will be tested at three different concentrations spaced to span the solubility range for each compound. The data for two test concentrations of each compound are the subject of this paper.

ACTIVATED SLUDGE PLANT DESIGN AND OPERATION

The main components in each system consisted of a completely-mixed, continuous flow reactor (15.2 cm I.D. cylinder) and two clarifiers (primary and secondary). See Figure 1. System components were composed of either glass (reactors, clarifiers), Teflon (tubing, connectors) or stainless steel (valves, covers) to prevent reactive interactions with the compounds being tested and to reduce the chance of sample contamination. The liquid volume of the reactor was 11 liters with a depth of 58.4 cm and a headspace volume of 5.7 liters. The clarifiers had liquid volumes of 1.5 liters with a headspace volume of 0.8 liters. The reactor was air-mixed (0.8 - 1.0 liter/min) using an air stone diffuser. All vessels were covered and the off-gasses were passed through either ventilation tubing or gas sampling traps at a rate of approximately 20-50 cc/min.

Peristaltic and syringe pumps, as well as gravity, were used to move the various liquid streams throughout the system. A peristaltic pump was used to deliver influent to each reactor at a flow rate of 11 liters per day, resulting in a 24 hour hydraulic retention time. The test compound was injected into the influent line using a syringe pump prior to a series of small diameter mixing loops and the primary clarifier. The primary effluent flowed by gravity to the reactor and the reactor contents overflowed by gravity to the secondary clarifier. Settled sludge was returned to the reactor from the secondary clarifier via a recycle pump on a timer at an average rate that ranged from approximately 11-22 mLs/min. Sludge was wasted directly from the reactor on a semi-continuous basis using a peristaltic pump operating intermittently at a high flow rate (23 mL every 30 minutes). The wasting rate for both systems was set at 10% of the reactor volume per day to produce a theoretical solids retention time (SRT) of 10 days.

A synthetic feed, using the recipe described by Kincannon (1983), was prepared daily and stored at 4 C while it was pumped to the system. A synthetic feed was chosen to facilitate comparisons between test runs with different compounds as well as to ensure a stable source of substrate and nutrients. The initial activated sludge seed was a return activated sludge from a larger activated sludge system operated at the Test & Evaluation Facility. This plant treats the same influent (approximately 40% industrial wastes) received at the Cincinnati Mill Creek Sewage Treatment Plant. On a daily basis, 100 mLs of primary effluent from this same larger T&E activated sludge system were added to each reactor to provide a continuing innoculum of microorganisms not found in the synthetic feed. This volume of primary effluent, however, did not significantly affect the chemical composition of the reactor contents (100 mLs into 11,000 mLs of reactor volume).

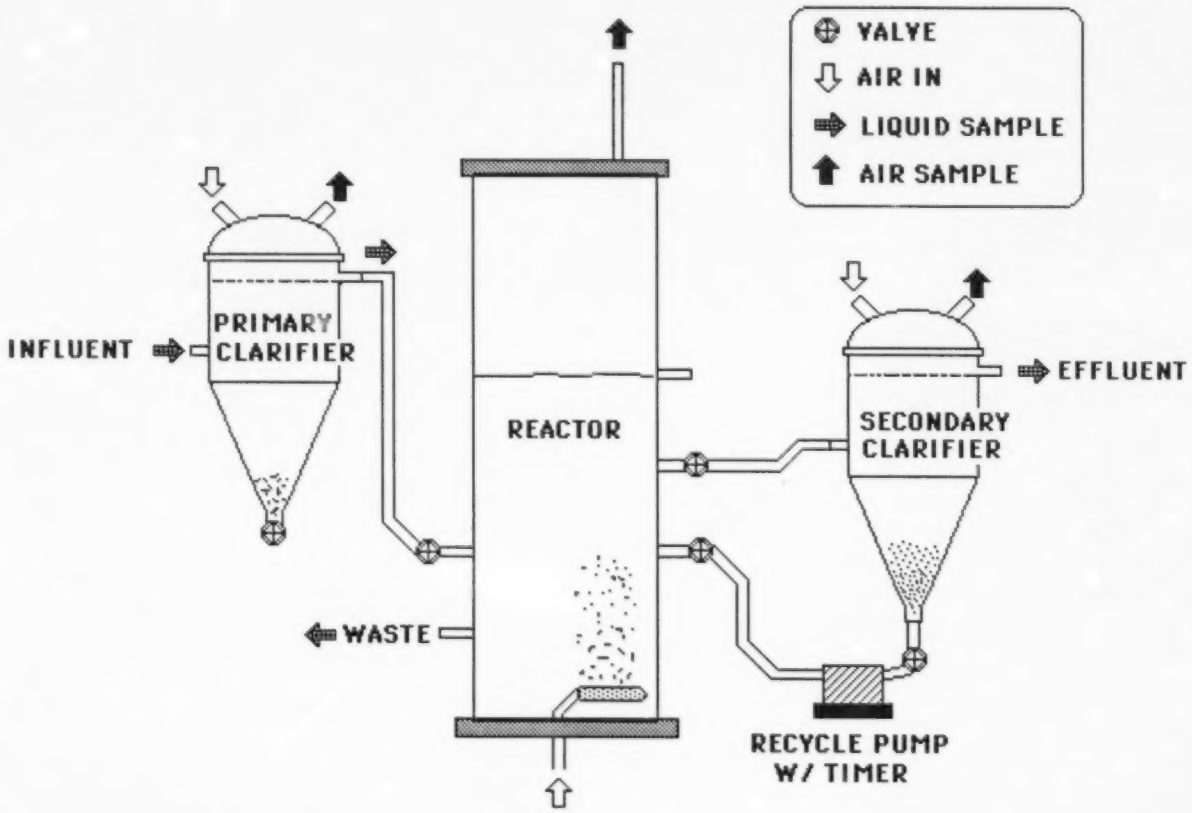

Figure 1. Activated Sludge System Schematic

Both test units were acclimated to the feed for at least one month (= 3 theoretical SRT's) prior to initiation of spiking. Following acclimation to the base synthetic feed, both units were acclimated to their initial test compound concentrations for a period of at least one month prior to test phase data collection.

The routine process monitoring measurements, their frequencies and the methods used are summarized in Table 1. Influent, effluent and wasted mixed liquor were continuously stored/collected in carboys in a refrigerator at 4 C, and all tests were run within 48 hours of sample collection. Soluble biochemical oxygen demand (SBOD) and soluble organic carbon (SOC) were run on influent (unspiked) and effluent samples taken from the feed and effluent carboys, respectively. Total and volatile suspended solids (TSS/VSS) were run on 24-hr composite wasted mixed liquor and effluent samples. Dissolved oxygen concentration (D.O.) was measured in-situ in each reactor at approximately four hour intervals. Dissolved oxygen uptake rate (DOUR) was measured on a grab sample of mixed liquor. This sample was returned to the reactor after testing to conserve solids in the reactor and maintain the calculated SRT.

Table 1. Routine Process Monitoring

Measurement	Frequency	Method
SBOD	3X/week	EPA 405.1
SOC	5X/week	EPA 415.2
TSS/VSS	1X/day	EPA 160.1
D.O.	6X/day	EPA 360.1
DOUR	1X/day	Standard Methods 213A

MEK/TCA SAMPLING AND ANALYSIS

Samples were collected and analyzed for the appropriate test compound three times per week for at least two weeks during the test phase. Influent and secondary effluent samples were continuously collected over a 24-hour period at a low flowrate (1.0 ml/min) in refrigerated Teflon bags with minimal headspace to reduce losses due to volatilization. Wasted mixed liquor samples were also continuously collected but in refrigerated 2-liter Erlenmeyer flasks because reactor concentrations of suspended solids clogged the Teflon bag valves. Prior to sample delivery, a portion of each composite was transferred to an amber glass vial and sealed using a Teflon-lined lid. The vial was filled completely (no headspace) to reduce volatile losses during transport and storage. Primary effluent samples were collected as grab samples from the line between the primary clarifier and the reactor. All samples were stored at 4 C prior to analysis and were analyzed within 48 hours of sample collection.

Primary clarifier, secondary clarifier, and reactor off-gases were sampled using 0.63 cm O.D. borosilicate glass traps each packed with 0.25 grams of Tenax. Two traps were used in series for each sample, with the second trap in place to quantitate any breakthrough that occurred. Air sampling pumps were used to supply the appropriate rate of air flow, which was further controlled by needle valves and measured by rotameters. Off-gas removed from the clarifiers was replaced by purified air at atmospheric pressure during sample collection. Ambient air was used at all other times. The amount of time required for gas sampling varied with the concentration of the test compound in the gas and with the detection limits of the analytical procedure. Once the gas samples were obtained, the traps were sealed and refrigerated at 4 C until analysis.

EPA Method 8015 (SW-846, 1986) was used to measure methyl ethyl ketone (MEK) and EPA Method 601 (Federal Register, 1984) was used to measure 1,1,1-trichloroethane.

DISCUSSION OF RESULTS

The following discussion of results has been divided into two sections. The first section presents the activated sludge process monitoring data for both reactors and includes experimental observations concerning operation of the reactors. The second section contains a description of the mass balance calculations used to identify the predominant removal mechanism for each test compound with a summary of the MEK/TCA results.

Activated Sludge Process Monitoring Data

Table 2 summarizes the routine process monitoring data for SBOD, SOC and TSS/VSS for the MEK system at two concentrations. Mean values with standard deviations for the two week period just before the test phase and for the two week test period itself are presented.

Table 2. Process Monitoring Data for MEK System
(Mean ± Standard Deviation in mg/L)

	MEK Concentration 1 55 ± 9 mg/L		MEK Concentration 2 430 ± 59 mg/L	
	Before Test	During Test	Before Test	During Test
Average SRT *	4.3	7.1	8.1	7.2
MLTSS	863 ±133	1210 ±378	2615 ±320	2368 ±194
MLVSS	779 ±121	1093 ±336	2430 ±298	2189 ±182
Effluent TSS	113 ± 69	50 ± 23	60 ± 30	93 ± 60
Effluent VSS	101 ± 62	44 ± 22	55 ± 30	84 ± 57
Influent SBOD	***	610 ±116	657 ±408	511 ± 39
Effluent SBOD	***	16 ± 8	33 ± 56	23 ± 19
% SBOD Removal	***	97	95	95
Influent SOC	420 ± 11	424 ± 8	421 ± 5	427 ± 9
Effluent SOC	38 ± 21	18 ± 6	10 ± 6	4 ± 4
% SOC Removal	91	96	98	99

* SRT in days
*** Quality control difficulties with SBOD analysis during this period of data collection.

Because a system upset occurred as a result of operational problems (sudden ambient/reactor temperature drop and mixer failure), a five-week period separated the second and third sample days during the testing of MEK at the first concentration. Once the problems were corrected, the system was allowed to re-equilibrate for approximately one month (= 3 theoretical SRT's) before testing resumed. In this case, the data presented in Table 2 for the period before testing represent the two weeks prior to the last five sample days. The data for the period prior to the first two sample days were comparable to these data with the exception of the mixed liquor total and volatile suspended solids concentrations (1970 ± 170 and 1625 ± 135 mg/L, respectively) and the effluent total and volatile suspended solids concentrations (22 ± 7 and 18 ± 12 mg/L, respectively). The distribution of MEK in the streams

leaving the reactor did not appear to be measurably affected by these differences, so the data from all of the sample days were used in the mass balance calculations discussed later.

The dissolved oxygen concentration averaged 4.7 mg/L (1.3-8.2 mg/L range) during the time the reactor was spiked with 55 mg/L MEK. The dissolved oxygen uptake rate averaged 23 mg/L-hr. During the time the reactor was spiked with 430 mg/L MEK, the D.O. concentration averaged 4.3 mg/L (1.1-8.0 mg/L range) and the DOUR averaged 30 mg/L-hr.

Table 3 summarizes the process monitoring data for SBOD, SOC, and TSS/VSS for the TCA system at two concentrations. The values presented for the second test period represent data for only three days of testing. The reactor exhibited signs of shock and inhibition when the TCA concentration was increased to the second concentration. Reactor solids became light and disperse and began washing out of the system in the effluent. The average SRT dropped from 6 days to less than 2 days. Limited sampling was initiated two weeks after the concentration was changed in order to examine the distribution of TCA even though the system was obviously not in equilibrium. The mass balance data for this sampling period is qualitatively but not quantitatively discussed later in the paper.

Table 3. Process Monitoring Data for TCA System
(Mean ± Standard Deviation in mg/L)

	TCA Concentration 1 141 ± 37 mg/L		TCA Concentration 2 174 ± 32 mg/L	
	Before Test	During Test	Before Test	During Test
Average SRT *	4.2	6.0	1.8	1.2
MLTSS	1048 ±162	1471 ±193	770 ±247	533 ± 23
MLVSS	958 ±129	1335 ±178	682 ±221	467 ± 29
Effluent TSS	140 ±119	100 ± 69	355 ± 66	392 ± 50
Effluent VSS	134 ±112	91 ± 65	327 ± 58	345 ± 75
Influent SBOD	485 ± 16	***	488 ± 16	501 ± 13
Effluent SBOD	17 ± 16	***	6 ± 4	10 ± 3
% SBOD Removal	96	***	99	98
Influent SOC	420 ± 10	426 ± 13	416 ± 8	425 ± 4
Effluent SOC	20 ± 12	27 ± 14	13 ± 8	8 ± 1
% SOC Removal	95	94	97	98

* SRT in days

*** Quality control difficulties with SBOD analysis during this period of data collection.

The dissolved oxygen concentration averaged 4.4 mg/L (2.0-6.8 mg/L range) during the time the reactor was spiked with 141 mg/L TCA. The dissolved oxygen uptake rate averaged 16 mg/L-hr. During the time the reactor was spiked with 174 mg/L TCA, the D.O. concentration averaged 5.4 mg/L (4.0-6.5 mg/L range) and the DOUR averaged 12 mg/L-hr.

The data in Tables 2 and 3 indicate that the average SRT was not maintained at the target value of 10 days. This was due in part to operational problems. Both the MEK and TCA systems experienced secondary sludge settling and recycle problems attributable to the character of the sludge formed and the design of the secondary clarifier. The sludge flocs formed were fine and light and did not settle well. Floc formation problems may be associated with the use of the synthetic wastewater. Nutrient deficiency may also have been a factor in the operation of the MEK system at the higher concentration. The amounts of nitrogen and phosphorus in the feed were not increased to match the increase in organic loading due to additional MEK during this phase of the study. They were adjusted when the amount of MEK spiked was raised to the third concentration.

The secondary clarifier design also affected the operation of the systems. The clarifier covers were removed and the sides and bottom conical portion of the clarifier were manually scraped once a day. However, the solids collecting in the bottom still had a tendency to "bridge" across the tip of the cone due to the diameter/slope ratio for the scale of the clarifier. The solids "bridge" then acted like a filter, retaining solids in the clarifier. Since the original clarifier design did not include baffles, this may have been compounded at times by short-circuiting between the influent and effluent ports. Both of these factors contributed to the somewhat high and variable TSS/VSS concentrations in the effluent. The clarifiers have since been modified to include a center well to prevent short-circuiting. More frequent scraping has been instituted to reduce the bridging problem, however, periodic manual scraping itself can create disturbances to the equilibrium of the system.

Aside from the solids control problems, both systems exhibited high SBOD and SOC removals throughout the study, indicating that the soluble compounds in the feed were readily removed from the system. However, this does not necessarily mean that the system, as operated in this study, would meet discharge criteria. The solids in the effluent have the potential to increase the total BOD of the effluent to unacceptable levels. The data obtained in this study cannot be used to judge how well a large scale activated sludge treatment plant would operate at the test compound concentrations studied. It can, however, be used to identify the removal mechanisms for the test compounds.

MEK/TCA Mass Balance Calculations and Results

In order to identify the predominant removal mechanisms for each test compound, mass balances were calculated around both the primary clarifier and the reactor/secondary clarifier. Tables 4 and 5 summarize this data for MEK. The TCA data are summarized in Table 6.

In the balance around the primary clarifier, the mass flux out of the clarifier in both the primary effluent and the off-gas should equal the mass flux into the clarifier in the influent. The difference in these terms is designated as "unknown primary losses/gains" and is probably due to analytical and sampling variations.

$$Mi - (Mpe + Mpg) = Mun \qquad (1)$$

where Mi = mg/L test cpd in influent x 0.0076 L/min;
Mpe = mg/L test cpd in primary eff. x 0.0076 L/min;
Mpg = mg test cpd in pri. clar. off-gas collected per min sample collection time; and
Mun = mg/min unknown primary losses/gains.

In the balance around the reactor/secondary clarifier, the mass fluxes out of the reactor in the wasted mixed liquor and the reactor off-gas plus the mass fluxes out of the secondary clarifier in the secondary effluent and the clarifier off-gas plus any losses due to "apparent biodegradation" should equal the mass flux into the reactor from the primary clarifier. In this case, the "apparent biodegradation" term may include losses due to chemical reaction, photolysis and/or hydrolysis in addition to actual bio-oxidation. Also, since it is calculated as the difference between influent and effluent mass fluxes, it may include unknown losses/gains due to analytical and sampling variations.

$$Mpe - (Mrw + Mrg + Mse + Msg) = Mab \qquad (2)$$

where Mpe = mg/L test cpd in primary eff. x 0.0076 L/min;
Mrw = mg/L test cpd in reactor waste x 0.00076 L/min;
Mrg = mg test cpd in reactor off-gas collected per min sample collection time x off-gas:sample volume ratio;
Mse = mg/L test cpd in secondary eff. x 0.0068 L/min
Msg = mg test cpd in sec. clar. off-gas collected per min sample collection time; and
Mab = mg/min apparent biodegradation.

In order to quantitate the relative amount of test compound distributed to each fate, the percentage of the test compound found in each stream was calculated. This was done by dividing the mean mass flux in the stream by the mean mass flux in the influent stream to the part of the system being considered. The overall removal of the test compound from the system was calculated by dividing the difference between the mean mass fluxes in the influent to the primary clarifier and the effluent from the secondary clarifier by the mean mass flux in the influent to the primary clarifier.

As the data in Tables 4 and 5 indicate, biodegradation appears to be a primary mechanism for removal of MEK in the activated sludge system at the concentrations studied. In general, MEK was not found in appreciable amounts in the gas samples, the wasted sludge samples or the effluent, with the exception of the reactor off-gas sample at the higher MEK spike concentration. As for the latter case, this may indicate that stripping could become an increasingly important removal mechanism at higher MEK concentrations. Research is currently being done on MEK at a third, higher concentration to test this possibility. The data in Tables 4 and 5 also indicate that a portion of the MEK was lost through volatilization in the primary clarifier prior to contact with the biomass.

Table 4. MEK Concentration 1 (55 ± 9 mg/L) Mass Balances

	Flux (mg/min)	Percent (%)
PRIMARY CLARIFIER		
Influent	0.414 (± 0.067)*	-
Effluent	0.404 (± 0.056)	97.6
Pri. volatilization	0.046 (± 0.008)	11.1
Unknown loss/gain	-0.036	- 8.7
REACTOR/SECONDARY CLARIFIER		
Reactor influent	0.404 (± 0.056)	-
Reactor waste	<0.0004 (± 0.0004)	<0.1
Reactor stripping	<0.012 (± 0.012)	<3.0
Clarifier effluent**	<0.009 (± 0.009)	<2.3
Sec. volatilization	<0.0002 (± 0.0002)	<0.1
Apparent biodegrad.	0.382	>94.5

* Mean (± standard deviation)
** Overall removal from system = >99%

Table 5. MEK Concentration 2 (430 ± 59 mg/L) Mass Balances

PRIMARY CLARIFIER	Flux (mg/min)	Percent (%)
Influent	3.269 (± 0.449)*	-
Effluent	3.358 (± 0.223)	102.7
Pri. volatilization	0.084 (± 0.055)	2.6
Unknown loss/gain	-0.173	- 5.3
REACTOR/SECONDARY CLARIFIER		
Reactor influent	3.358 (± 0.223)	-
Reactor waste	<0.0004 (± 0.0004)	<0.1
Reactor stripping	0.342 (± 0.325)	10.2
Clarifier effluent**	<0.001 (± 0.001)	<0.1
Sec. volatilization	<0.003 (± 0.003)	<0.1
Apparent biodegrad.	3.012	>89.6

* Mean (± standard deviation)
** Overall removal from system = >99%

The data in Table 6 indicate that volatilization from the primary clarifier and stripping from the reactor are more important removal mechanisms than biodegradation for TCA. This is not surprising since the volatility of TCA made it difficult to keep this compound in solution during spiking and sampling procedures. Theoretical spike additions of 295 mg/L and 705 mg/L resulted in measured influent concentrations of 141 ± 37 mg/L and 174 ± 32 mg/L during the first and second test periods, respectively.

Kincannon et al. (1983) found that with an influent concentration of 118 mg/L TCA, stripping accounted for 100% of the removal, with 1.6 mg/L TCA remaining in the effluent. The average TCA concentration in our effluent samples was 1.3 ± 0.4 mg/L (0.010 ± 0.003 mg/min / 0.0076 L/min) at an influent concentration of 141 ± 37 mg/L, and stripping accounted for at least 76% of the removal from the reactor. The limited sampling at an influent concentration of 174 ± 32 mg/L did not allow calculation of a meaningful mass balance. However, the concentration of TCA in the effluent averaged 3.3 ± 0.4 mg/L and volatilization/stripping again appeared to be the primary removal mechanism. TCA can be biodegraded, as shown by Tabak et al. (1981). Given the extended acclimation period for the biomass in this system (one month at 10 mg/L and three months at 141 mg/L), some loss of TCA due to biodegradation was plausible, but it did not appear to be as effective a removal mechanism as volatilization/stripping at the concentrations studied.

Table 6. TCA Concentration 1 (141 ± 37 mg/L) Mass Balances

PRIMARY CLARIFIER	Flux (mg/min)	Percent (%)
Influent	1.072 (± 0.281)*	-
Effluent	0.741 (± 0.074)	69.0
Pri. volatilization	0.221 (± 0.066)	20.6
Unknown loss/gain	0.110	10.4
REACTOR/SECONDARY CLARIFIER		
Reactor influent	0.741 (± 0.074)	-
Reactor waste	0.001 (± 0.0002)	0.1
Reactor stripping	0.565 (± 0.218)	76.2
Clarifier effluent**	0.010 (± 0.003)	1.4
Sec. volatilization	0.004 (± 0.002)	0.5
Apparent biodegrad.	0.161	21.8

* Mean (± standard deviation)
** Overall removal from system = 99%

CONCLUSIONS

The primary removal mechanism for MEK in an acclimated activated sludge system at the concentrations studied (55 ± 9 mg/L and 430 ± 59 mg/L) appears to be biodegradation. In addition, stripping may become an increasingly important removal mechanism as the concentration of MEK is raised further. This phenomenon is currently being investigated. The effect of the studied concentrations on the operation of a full scale activated sludge plant is not clear as a result of solids settling/return problems experienced during this phase of the study.

The primary removal mechanism for TCA appears to be volatilization/stripping. Some biodegradation was evidenced, but it is not likely to be an important removal mechanism, especially at higher concentrations, due to TCA's inhibitory effects on biomass growth.

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